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METHODS FOR SAMPLING OF POWDERS FOR POWDER METALLURGICAL PURPOSES

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Indian Standard

METHODS FOR SAMPLING OF POWDERS FOR POWDER METALLURGICAL PURPOSES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 February 1972, after the draft finalized by the Methods of Sampling Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Metal powders find a wide variety of use, as in welding, manufacture of engineering components, electronics, chemical and metallurgical industries. It is estimated that more than two-thirds of all metal powders are used in the manufacture of powder metallurgy parts. Powder metallurgical processes permit the production of metals with physical properties different from those of metals prepared by more conventional methods. The powders most commonly used in conventional powder metallurgy are those of iron and iron base alloys such as stainless steel, copper and copper base alloys such as brass and bronze, nickel and nickel base alloys, lead, cobalt, tungsten, aluminium and titanium. There are non-metallic powders also such as of silicon and carbon which are widely used in powder metallurgy.

0.3 The rapid progress, made in recent times in the field of powder metallurgy in a number of countries, has necessitated that the practices followed in this country be co-ordinated with those established at the international level to facilitate trade and technology in the field. Sampling being an important aspect in the transaction of manufactured products, the need has arisen for laying down national standard on this subject.

0.4 In the preparation of this standard considerable assistance has been derived from ISO/TC 119 (Secretariat 61) 85 E ' Draft ISO proposal on sampling of powders for powder metallurgical purposes '.

0.5 In reporting the result of a test or analysis, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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1. SCOPE

1.1 This standard prescribes the methods for sampling of powders for powder metallurgical purposes and covers the reduction of the selected samples into the quantity required for testing for the determination of particle size,

*Rules for rounding off numerical values (*revised*).

moisture content, chemical composition, flow rate, compressibility, etc. The methods covered in this standard are primarily applicable to metallic powders.

1.2 This standard also lays down a method of reporting the quality of the material sampled.

2. TERMINOLOGY

2.0 For the purpose of this standard the following terms shall apply.

2.1 Lot — Consignment or a portion thereof comprising powder from the same batch of manufacture packed in one size of containers.

2.2 Increment — The amount of powder removed by a sampling device at one time.

2.3 Gross Sample — A blend of all the increments taken from a single inspection lot.

2.4 Analysis Sample — The amount of powder taken from the gross sample after necessary reduction and adequate for carrying out the required tests.

NOTE 1 — If several analysis samples are required, they shall be taken simultaneously so that losses occurring in connection with handling and milling (if undertaken) can be kept as low as possible.

NOTE 2 — If milling is included in the preparations for testing, the quantity that is milled shall be sufficiently large so that the analysis sample may be obtained by proper reduction of quantity of the milled powder.

3. NUMBER OF INCREMENTS

3.0 As far as practicable, it is recommended that the sampling should be done when the powder is in motion, that is, during the discharge of the powder in a continuous stream (*see* **3.1**). When the lot is presented in the form of filled containers or packages, it shall be sampled in accordance with **3.2**.

3.1 Sampling During Discharge in Continuous Stream — If the entire inspection lot is discharged in a continuous stream through an opening it may be sampled during discharge. In such case, increments should be taken at regular intervals during the entire discharge period. The number of increments shall depend on the accuracy desired but would be limited by the facilities for handling the gross sample. It is recommended that a minimum of 30 increments be drawn so as to obtain a representative gross sample from the lot.

3.2 Sampling from Powders Packed in Containers — Unless otherwise specified, the number of containers listed below shall be selected from

the inspection lot when samples are taken from powders packaged in containers. For ensuring the randomness of selection of the containers in the lot, the applicable procedure detailed in IS : 4905-1968* may be followed. From every selected container a minimum of three increments shall be collected to provide a gross sample of a size suitable to represent the entire lot.

No. of Containers in the Lot	*No. of Containers from Which Samples Should be Taken*
Up to 50	3
51 ,, 100	4
101 ,, 150	5
151 ,, 300	7
301 and above	10

4. SAMPLING

4.1 General — Samples shall be taken in such a manner that they represent the lot as accurately as possible and do not deviate from its average quality. All the surfaces of sampling devices, which come into contact with the powder, shall be smooth. All equipment and containers shall be clean and preferably made of stainless steel.

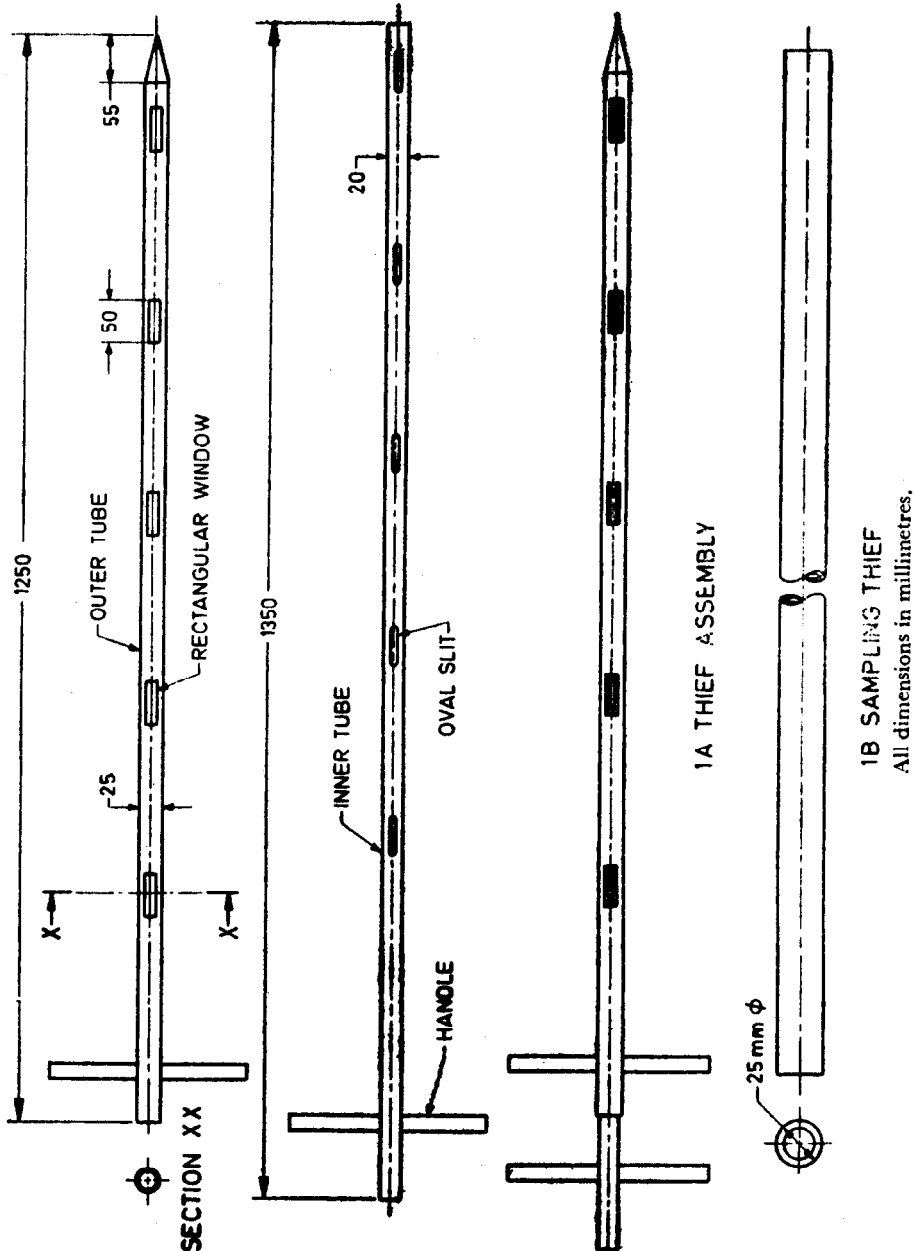
NOTE — Segregation may occur at any time when a batch of powder is set in motion that is, when filling and emptying containers, the shaking occurring during transportation and vibrations occurring during storage. Segregation risks increase with the flow rate of the powder. In addition, atmospheric conditions can cause local variations in moisture content and oxygen content to cite two examples.

4.2 Sampling During Discharge in Continuous Stream — The sampling container shall be rectangular and sufficiently large so that the increment taken does not fill it. Its dimensions at right angles to the stream of powder shall exceed the cross section of the stream by an ample margin. Moreover, the top of the container shall be of symmetrical design.

The sampling container shall be moved directly across the stream of powder at a constant speed. The speed shall be selected with regard to the desired size of the increments.

4.3 Sampling with a Sampling Thief — Different types of sampling thieves shall be used for powder with high and low flow rates. The design details depend on the powder from which the sample is to be taken. Line sketches of two types of sampling thieves commonly used are given in Fig. 1.

*Methods for random sampling.



All dimensions in millimetres.

4.3.1 For powder with a high flow rate, sampling thieves comprising an outer tube with closed end and longitudinal slip-shaped windows running the entire insertion length shall be used. Within the outer tube there shall be an inner tube also provided with slip-shaped windows positioned so that they are opened and closed successively as the tubes are twisted relative to each other. The tubes shall preferably be made of stainless steel and shall fit each other loosely enough so that twisting is not hindered by the largest particles encountered [*see* Fig. 1 (a)].

4.3.2 The sampling thief shall be inserted all the way to the bottom of the container with closed windows. It is recommended that it may be inserted in the direction which was vertical during transportation and storage. When the thief has reached the bottom, the windows shall be opened so that the thief is filled from bottom to top, after which the windows shall be closed and the thief withdrawn. The contents of the thief shall be emptied into the bulk sample container.

4.3.3 For powder with a low flow rate, sampling thieves comprising a tube with open end shall be used [*see* Fig 1. (b)]. The tube should be preferably of stainless steel and its diameter shall be selected so that all the powder forced into the tube, when it is inserted into the powder, remains there when the tube is withdrawn.

4.3.4 The sampling thief shall be inserted slowly all the way to the bottom of the container. It is recommended that it may be inserted in the direction which was vertical during transportation and storage. When the thief has reached the bottom, it shall be withdrawn and its contents emptied into the bulk sample container.

NOTE — If segregation has occurred in the direction in which the thief is inserted, errors will occur if the thief does not withdraw an equal quantity from every stratum.

4.3.5 To reduce the effects of segregation at right angles to the direction of thief insertion, the points of insertion shall be distributed as representatively as possible. If the inspection lot is, for example, packaged in identical cylindrical containers, the contents of the containers shall be divided into a number of tubular sections of the same width and increments shall be spread over as many different sections as possible.

5. REDUCTION OF SELECTED SAMPLE

5.1 General — The device for reduction of selected samples shall be of the proper size for the batch being split so that handling losses may be disregarded.

5.2 Quartering — All types of powder may be reduced by quartering.

5.2.1 A conical pile of the powder shall be poured into a clean and flat underlay, the powder always being added to the top of the pile. It is

recommended that the powder runs down through a funnel held in a fixed position relative to the underlay.

5.2.2 After all the powder is piled as described, the pile shall be flattened to about one-fourth of its original height by pressing it straight down with a flat plate. The plate shall be larger than the top surface of the pile after it has been depressed. The pile shall then be divided into four sections using a rigid perpendicular cross made of sheet metal. The arms of the cross shall be longer than the diameter of the pile. The cross shall be positioned with its centre on the centre of the pile and pressed down through the powder to the underlay. The powder in two opposite quadrants shall be collected and used for testing or, if further reduction is desired, the process shall be repeated.

5.3 Sample Splitter — Powder with a high flow rate can be split with a sample splitter. The powder shall be poured through a wide trough provided with a number of discharge openings that lead alternately to collection containers on both sides of the sample splitter (the even discharge openings lead to one side and the odd openings to the other). The powder collected in one collection container shall be used for testing or, if further reduction is desired, the process shall be repeated.

5.3.1 Sample splitters may only be used for batches of powder with a volume more than one-fifth of the trough volume or for batches of powder in which a maximum of 0·5 percent sticks to the sample splitter after being passed through it.

6. NUMBER OF TESTS

6.1 If it is intended to know only the average quality of the lot, then all the increments should be mixed, homogenized and reduced by any of the methods given under **5**. The characteristics shall then be determined on the composite sample so obtained.

6.2 If, however, it is intended to ascertain the uniformity of the various properties also, then the number of tests shall be as given in **6.2.1** and **6.2.2**.

6.2.1 In the case of sampling during discharge (*see* **3.1** and **4.2**) in a continuous stream, then every 5 increments in the order of drawing shall be mixed together and reduced separately. Thus there would be a minimum of 6 representative samples from a lot and these shall be analyzed individually.

6.2.2 In the case of sampling from containers (*see* **3.2** and **4.3**) the increments from each container shall be mixed separately and reduced. The properties shall then be determined on each of the individual samples representing the selected containers separately.

7. REPORTING

7.1 For those characteristics where a composite sample has been tested (*see* **6.1**), only one test result will be available and that result shall be reported as the value of the characteristic for the lot sampled.

7.2 When three or more laboratory samples have been analyzed individually from a lot for any characteristic (*see* **6.2**), the following procedure shall be followed to assess the average quality and its limit of variation:

Let $x_1, x_2, x_3, \ldots\ldots, x_n$ be the results of analyzing n laboratory samples for a particular characteristic.

Then calculate, average $\bar{x} = \frac{x_1 + x_2 + \ldots\ldots x_n}{n}$, and

Range R = difference between the maximum and the minimum values.

The average level of that characteristic in the lot shall be reported as equal to $\bar{x}$.

The limits of variation in the average level of the lot shall be reported as $(\bar{x} \pm hR)$, where h is a factor the value of which depends upon the number of samples analyzed. The appropriate value of the factor h shall be taken from the following:

No. of Laboratory Samples Analyzed	*Value of the Factor* h
3	1·30
4	0·72
5	0·51
6	0·40
7	0·33
8	0·29
9	0·25
10	0·23

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